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An Investigation of the System: Ferric Chloride-Hydrochloric Acid-Water-Isopropyl Ether

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An experimental investigation of the heterogeneous system: ferric chloride-water-hydrochloric acid-isopropyl ether at 20°C is reported. The change in volumes of the phases as a function of the quantity of each of the added components is determined. The equilibrium composition of the phases of the four-component system at various initial concentrations of hydrochloric acid and ferric chloride is analytically determined. From the results, orthogonal projections of the points representing the composition of the phases in a tetrahedral model are drawn. The results of the analyses and transient conditions from two-phase to three-phase systems (and *vice versa*) are discussed.

INTRODUCTION

The extraction of ferric chloride from an aqueous solution of hydrochloric acid by isopropyl ether has been the subject of several investigations. It is generally accepted that iron(III) is extracted as tetrachloroferric acid $HFeCl_4$[1,2] where the proton is hydrated[3]. It is also known that the distribution ratio of iron depends on its total concentration[4]. Explanations of this result based on various physico-chemical investigations are given[5-9]. It is also observed that at higher initial acid concentrations (above 7 *M* HCl) two ether phases are formed[4,6]. The appearance of the third phase is observed in several other extraction systems[10-14]. The formation of the third phase is of special interest to the separation because the concentration of the metal is always higher in the third phase than in the light ether phase. It is interesting to determine the initial conditions for which the third phase can be observed depending on the content of water, hydrochloric acid and iron in both ether phases.

In this paper is given a complete analysis of the phases in the two- and three-phase regions of a four-component system. This makes it possible to draw the orthogonal projections of points representing the composition of the phases in a 3-dimensional model.

EXPERIMENTAL

Materials

Isopropyl ether, *Fluka*, cp., was purified by treatment with saturated ferrous sulfate solution followed by sodium hydroxide solution. After washing with water,

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* Taken in part from the M. Sc. thesis of D. Maljković submitted to the Faculty of Science, University of Zagreb.

it was distilled and the middle fraction (b.p. 66.8° — 67.8°C, at 743.5 mm. Hg) was collected. Isopropyl ether prepared in this way contained 1.63 mg. H_2O/ml. The hydrochloric acid used was *Merck,* p. a. (d_4^{20} = 1.16). Anhydrous ferric chloride, *T. Schuchardt,* was 98 per cent pure. Other chemicals used were of analytical reagent grade.

Methods of analyses

Isopropyl ether, iron and chloride in the aqueous phase were determined by direct analysis. In the etherial phase, water, iron and chloride were determined directly. The fourth component in each phase was determined as the difference. Iron was determined for quantities less than 1 *mM* by UV spectrophotometric measurements and for larger quantities by complexometric titration (Titriplex III with 2-oxi-5-sulfo-benzoic acid[15]). A constant spectrum of ferric chloride was achieved by adding hydrochloric acid to a fixed concentration (2.54 *M*)[16]. The obtained regression of concentration of ferric chloride upon the absorption (measured at 335 mμ) is linear in the range between 1.1×10^{-3} to 5.0×10^{-5} *M* ferric chloride. If larger quantities were present, iron was determined by complexometric titration in the aqueous extract of the ether phase sample. For the analysis of chloride with $AgNO_3$, three titration methods (potentiometric, conductometric and volumetric methods with dichlorfluorescein as indicator) were tested. It was found that the most suitable method is potentiometric titration. The chloride samples of the aqueous phase were determined directly. The content of chloride in the ether phases was determined in the aqueous solution obtained by re-extraction of the sample. Water in the ethereal phase was analysed by the Karl Fischer[17] titration method modified by Laurene[18]. The dead-stop end point titration was used. For the analysis of ether in the aqueous phase the method proposed by Tuck[19] was applied. Centrifuge tubes (total volume 7 or 12 ml.) with a narrow neck were used. The necks were calibrated by measuring the increase in the height of the liquid in the neck (measured by cathetometer) due to the addition of a known amount of water. The zero correction, *i.e.* the solubility of isopropyl ether in the final solution in 7 ml. tubes was 9.27 mg. and in 12 ml. tubes was 19.57 mg. The ether column in the neck was formed by neutralization with sodium hydroxide, and by centrifuging (4000 rpm) the samples. All readings were made when a stable temperature (20°C) in the tube was attained. When larger quantities of iron were present, the determination was more difficult due to the precipitation of iron hydroxide. The ratio of the volumes of the equilibrium phases in a graduated separation funnel was determined with an accuracy of ± 0.05 ml. Most of the density determinations were made with 10 ml. pycnometers. Smaller volumes of the phases were determined by weighing the aliquote in a ground neck tube. Viscosity determinations of the third phase were carried out with an Ostwald viscometer having a measuring range between 2.0 and 4.0 cP.

Procedure

The systems were prepared in separation funnels. The funnels were shaken vigorously for 15 seconds and placed in a water bath thermostat at 20.0 ± 0.05°C. The shaking was repeated in the same manner, at fifteen-minute intervals, for one hour. We have found from experience that one hour is sufficient to reach the equilibrium, and this is in agreement with other reported work[1]. The determination of the changes in volume as a function of the quantity of each of the component in the system was made at room temperature (about 20°C). Titrations with anhydrous ferric chloride in a separation funnel were carried out using neck tubes specially adapted for bringing the material into the system. The funnels were shaken vigorously for one minute and the separation time was only 15 minutes.

Apparatus

The separations were made in 25 ml. and 50 ml. separation funnels having a calibrated stem. The determinations of viscosity and equilibration of the systems were performed by thermostat *Towson & Mercer,* Croydon at 20.00 ± 0.05°C. Measurements of ultraviolet absorption were made on a *Hilger* Uvispek spectrophotometer using quartz cells of 0.5 cm. Electrometric Karl Fischer titrations were made with a *Baird & Tatlock,* London instrument. Before measuring the content of ether in the aqueous phase the samples were centrifuged at 4,000 rpm for 5 minutes in a *Servall* centrifuge. The measurement of the ether column height was made with a *PYE-Cambridge* cathetometer. For potentiometric titrations, a pH-meter 4 *Radiometer* with Hg(I)/Hg_2SO_4 and Ag/AgCl electrodes was used.

RESULTS

Special attention is paid to the concentration region in which two or three liquid phases coexist. This region is near to the tetrahedron edge made by the triangular planes representing the systems: HCl-H_2O-IPE[20] and $FeCl_3$-H_2O-IPE[21].

Relations between volumes of phases

The relationships between the volumes of the equilibrium phases as a function of the quantity of each of the components were roughly determined at room temperature. This was done rarely for a guide. The volume of added hydrochloric acid (and the corresponding initial concentration of acid in the aqueous phase) vs. the volumes of the equilibrated phases are plotted in Fig. 1.

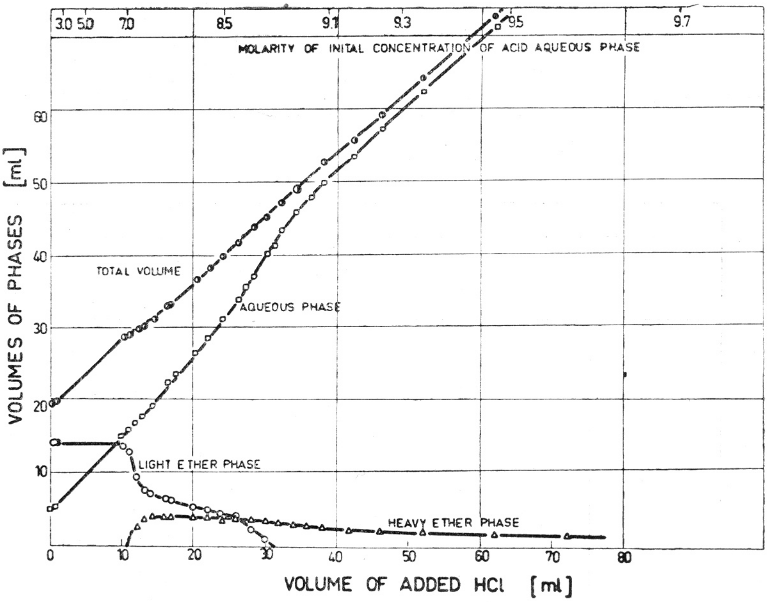

Fig. 1. Relationship between the volumes of the liquid phases. Titration of the system: $FeCl_3$-HCl-H_2O-IPE with hydrochloric acid (d_4^{20} = 1.161) at room temperature. Initial: 0.375 g. $FeCl_3$, 10.859 g. IPE and 4.971 g. H_2O.

At an initial concentration of hydrochloric acid in aqueous phase above 7 *M*, the third phase (heavy ether phase) is formed. The increase in the volume of the third phase corresponds to the decrease in the volume of the light ether phase. The third phase volume tends to a constant value. The influence of an added quantity of ferric chloride (anhydrous) on the ratio of the volumes of the phases is shown in Fig. 2. The increase of the content of ferric chloride in the quarternary system results in a decrease in volume of the aqueous phase due to the increase in the quantity of coextracting water. The volume

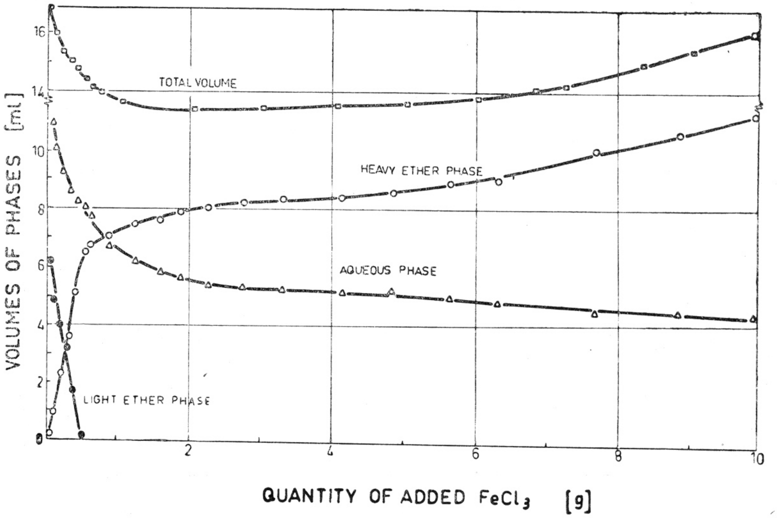

Fig. 2. Relationships between the volumes of the liquid phases. Titration of the system: $FeCl_3$-HCl-H_2O-IPE with anhydrous ferric chloride at room temperature. Initial: 3.002 g. HCl, 7.283 g. H_2O and 6.532 g. IPE.

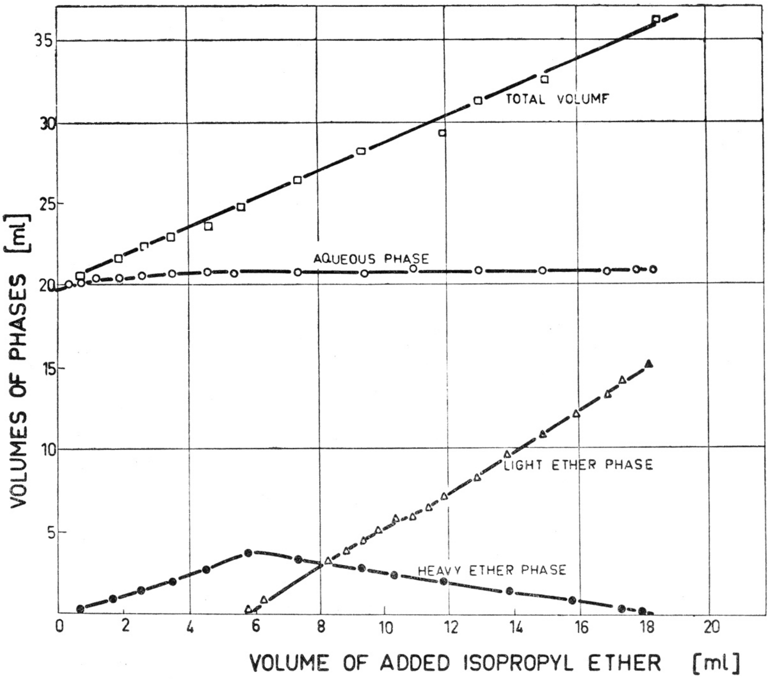

Fig. 3. Relationships between the volumes of the liquid phases. Titration of the system: $FeCl_3$-HCl-H_2O-IPE with isopropyl ether at room temperature. Initial: 0.214 g. $FeCl_3$, 5.630 g. HCl and 16.739 g. H_2O.

of the light ether phase decreased with an increase of ferric chloride and is inversely proportional to the volume of the third phase. After disappearance of the light ether phase the volume of the third phase was slightly increased further. The minimum of the total volume occured between the addition of 2 and 3 grams of ferric chloride. The influence of the isopropyl ether content is shown in Fig. 3. The third phase is formed prior to the formation of the light ether phase because the initial concentration is higher than 7 *M* hydrochloric acid and above 0.025 *M* ferric chloride. The quantity of isopropyl ether present in the system should exceed its solubility in the water phase before forming any of the ether phase. The light ether phase occured for the first time when the quantity of added isopropyl ether corresponded to the maximum value of the third phase volume. The position of the maximum is shifted with an increase of ferric chloride to the higher values of the volumes of added isopropyl ether. It seems that the quantity of the third phase depends (if the concentration of hydrochloric acid is sufficient) on the quantity of ferric chloride. Also, Table I shows the position of disappearance of the third phase.

TABLE I

Position of the Maximum Volume of the Third Phase and its Disappearence During the Titration.
(Constant Composition: 5.63 g. HCl and 16.76 g. H_2O)

$FeCl_3$ g.	ml. IPE where the maximum volume of the third phase occurs	ml. IPE where the third phase disappears
0.071	4.0	8.0
0.214	5.8	18.0
0.357	8.0	33.0

The third phase disappears when the concentration remains below 7 *M* hydrochloric acid in the aqueous phase. In Fig. 4 the volumes of the phases are plotted against the volume of water. The initial composition of the system is chosen so as to assure the formation of all three phases. Very interesting results are obtained by further addition of water. The volumes of both ethereal phases increase, however, the volume of the aqueous phase decreases. When the quantity of water added to the system is sufficient to dilute the hydrochloric acid below the critical concentration, the third phase suddenly disappears and the volume of the light ether phase shows a corresponding increase. There is no difference in the UV and visible absorption spectra for the two ether phases[3]. In the two-phase region (aqueous phase with light ether or heavy ether phase) it is necessary to distinguish between the ethereal phases. This can be achieved by viscosity or density measurements. The viscosity of the third phase varies between 2 and 4 cP and that of the light ether phase is about 0.6 cP.

Equilibrium composition of liquid phases

The equilibrium composition of the coexisting liquid phases was determined within two series of systems by varying the initial concentration of ferric chloride (from 0.05 to 2.96 *M*) and hydrochloric acid (from 2.47 to 10.29 *M*). The ratio of the aqueous solution to ether is 1.25 : 1 for all systems. The compositions of the liquid phases for various initial concentrations of ferric chloride and hydrochloric acid are given in Tables II and III respectively. The results obtained by the analyses give the co-ordinates of the points in the tetrahedral model which represent the composition of the coexisting equilibrium liquid phases. In Figs. 5—8 the orthogonal projections of these points onto the planes representing the ternary systems: $FeCl_3$-H_2O-IPE and HCl-H_2O-IPE are shown. These diagrams qualitatively indicate the position of the third phase in the tetrahedral model, but however, for a complete definition of the third phase many analyses are required. Graphical interpolation of the tie lines is not convenient in this case as in the ternary system.

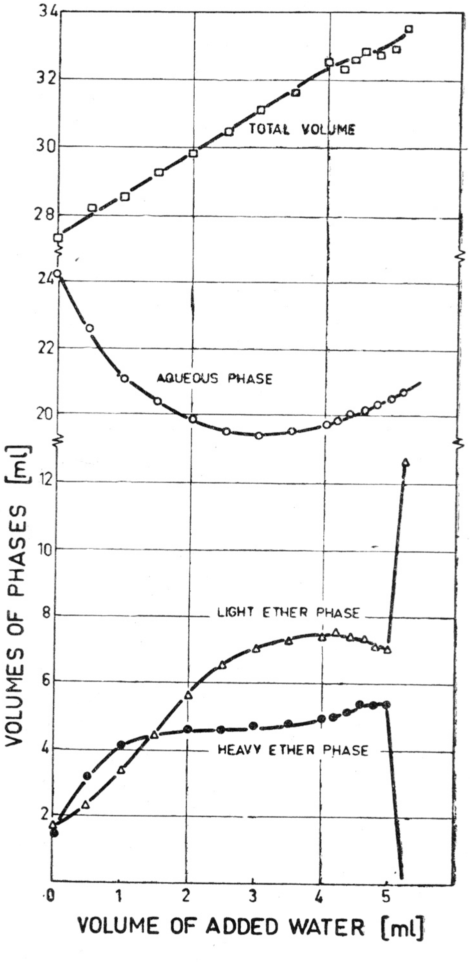

Fig. 4. Relationships between the volumes of the liquid phases. Titration of the system: $FeCl_3$-HCl-H_2O-IPE with water at room temperature. Initial: 11.784 g. H_2O, 5.630 g. HCl, 0.389 g. $FeCl_3$ and 10.859 g. IPE.

TABLE II

Molarity of Components of Equilibrium Phases in the System: $FeCl_3$-HCl-H_2O-Isopropyl Ether at 20°C for Various Initial Concentrations of Ferric Chloride (Initial: 8.23* M *HCl and Ratio $H_2O/IPE = 1.25 : 1$)*

Initial concn. $FeCl_3$	Aqueous phase				Heavy ether phase				Light ether phase			
	$FeCl_3$	IPE	H_2O	HCl	$FeCl_3$	IPE	H_2O	HCl	$FeCl_3$	IPE	H_2O	HCl
0.05	0.00	0.30	45.24	7.32	0.62	6.23	4.20	0.81	0.05	6.92	0.33	0.06
0.10	0.00	0.29	44.82	7.43	0.54	6.34	3.77	0.74	0.05	6.95	0.30	0.05
0.17	0.00	0.27	45.22	7.30	0.53	6.37	3.36	0.66	0.06	6.99	0.33	0.07
0.40	0.00	0.27	45.75	7.53	0.62	6.54	3.36	0.72	—	—	—	—
0.62	0.00	0.25	45.40	7.30	0.81	6.44	4.07	1.04	—	—	—	—
0.99	0.01	0.21	46.07	7.21	1.18	5.58	4.52	1.07	—	—	—	—
1.48	0.10	0.21	47.13	7.01	1.50	6.02	5.06	1.30	—	—	—	—
2.22	0.52	0.21	45.76	6.70	1.81	5.82	5.46	1.26	—	—	—	—
2.96	1.05	0.21	44.94	6.30	1.99	5.60	5.68	1.63	—	—	—	—

* IPE — ispropyl ether

TABLE III

Molarity of Components of Equilibrium Phases in the System: $FeCl_3$-HCl-H_2O-Isopropyl Ether for Various Initial Concentrations of Hydrochloric Acid at 20°C. (Initial: 0.197 M *$FeCl_3$ and Ratio $H_2O/IPE = 1.25 : 1$)*

Initial concn. HCl mol.	Aqueous phase				Heavy ether phase				Light ether phase			
	$FeCl_3$	IPE	H_2O	HCl	$FeCl_3$	IPE	H_2O	HCl	$FeCl_3$	IPE	H_2O	HCl
10.29	0.03	2.11	32.30	6.60	0.86	4.43	18.34	2.27	—	—	—	—
9.88	0.03	1.58	36.00	6.68	0.62	4.72	9.85	2.37	—	—	—	—
9.06	0.00	0.70	40.70	7.96	0.62	5.66	5.74	1.43	0.03	6.52	0.06	0.17
8.65	0.00	0.53	42.29	7.76	0.59	5.78	4.65	1.05	0.05	6.93	0.30	0.05
7.82	0.00	0.24	45.42	7.24	0.46	6.70	1.06	0.56	0.08	6.89	0.56	0.11
6.18	0.01	0.08	48.12	5.96	—	—	—	—	0.31	6.72	2.27	0.29
4.94	0.07	0.05	49.81	4.65	—	—	—	—	0.21	6.81	1.56	0.17
3.75	0.16	0.05	50.76	3.71	—	—	—	—	0.01	7.03	0.28	0.01
2.47	0.20	0.05	52.00	2.46	—	—	—	—	0.00	7.06	0.16	0.00

The results in Table VI show that a large portion of iron which is extracted is present in the third phase. The third phase, when separated from other phases of various compositions and kept at room temperature (the samples being closed in stoppered tubes) did not decompose after six months, indicating great stability.

DISCUSSION

The third phase is formed at the expense of the light ether phase as is seen from Fig. 1 and 2. If the system is titrated with hydrochloric acid an increase in the solubility of ether in the aqueous phase contributes also to the decrease of the volume of the light ether phase. The observed interaction of the phases (the minimum of the total volume, Fig. 2) indicates an intensified coextraction of water which is very closely bonded in the extracted and hydrated species forming the third phase. The increase in the volume of the light ether phase and the decrease in the volume of the aqueous phase by addition of water to the system (Fig. 4) can be partially understood by a reduction in the solubility of ether in the aqueous phase due to a decrease in

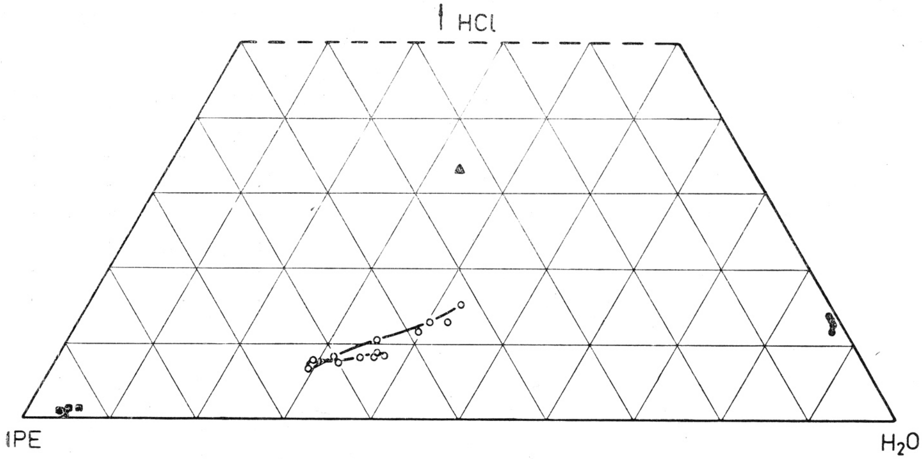

Fig. 5. Orthogonal projections of points representing the composition in molar per cents of the equilibrium liquid phases in the four-component extraction system at 20°C for various initial concentrations of $FeCl_3$ (calculated from the results of Table II). The plane of projection is the three-component system HCl-H_2O-IPE.

- ● aqueous phase
- ○ heavy ether phase
- ■ light ether phase
- ▲ projection of the top of the tetrahedron representing the pure fourth component.

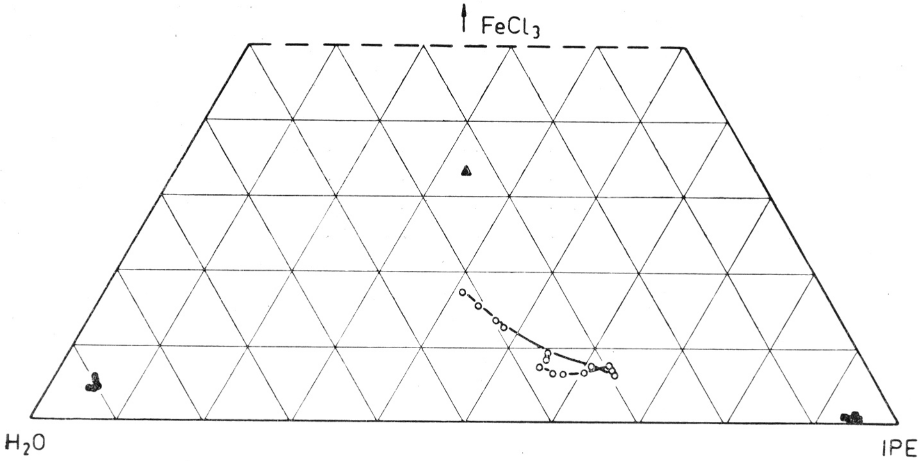

Fig. 6. Orthogonal projections of points representing the composition in molar per cents of the equilibrium liquid phases in the four-component extraction system at 20°C for various initial concentrations of $FeCl_3$ (calculated from the results of Table II). The plane of projection is the three-component system $FeCl_3$-H_2O-IPE.

- ● aqueous phase
- ○ heavy ether phase
- ■ light ether phase
- ▲ projection of the top of the tetrahedron representing the pure fourth component.

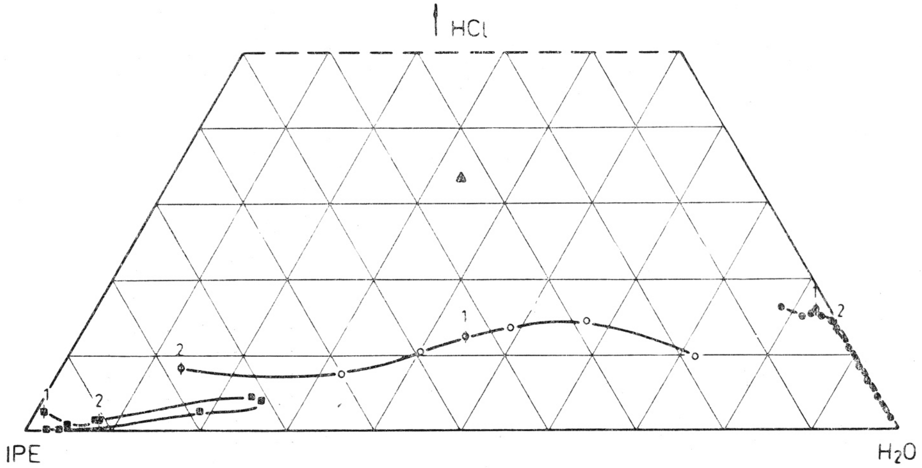

Fig. 7. Orthogonal projections of points representing the composition in molar per cents of the equilibrium liquid phases in the four-component extraction system at 20°C for various initial concentrations of HCl (calculated from the results of Table III). The plane of projection is the three-component system HCl-H_2O-IPE.

- ● aqueous phase
- ○ heavy ether phase
- ■ light ether phase
- ▲ projection of the top of the tetrahedron representing the pure fourth component.

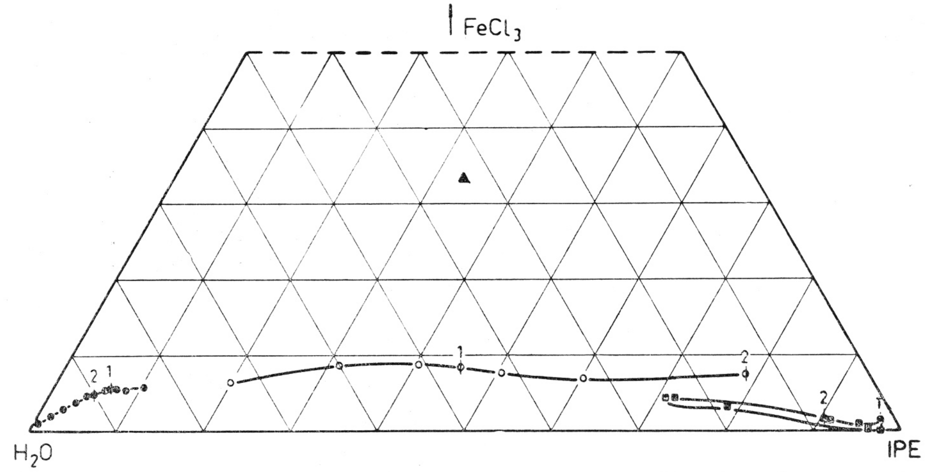

Fig. 8. Orthogonal projections of points representing the composition in molar per cents of the equilibrium liquid phases in the four-component extraction system at 20°C for various initial concentrations of HCl (calculated from the results of Table III). The plane of projection is the three-component system $FeCl_3$-H_2O-IPE.

- ● aqueous phase
- ○ heavy ether phase
- ■ light ether phase
- ▲ projection of the top of the tetrahedron representing the pure fourth component.

its acidity. The increase in the volume of the third phase may be due to the less dehydrating influence of the aqueous phase. A consequence would be the increase in the hydration number of the etherate of tetrachloroferric acid to five[3]. The etherate of tetrachloroferric acid then becomes soluble in the isopropyl ether and results in a decrease in volume of the third phase.

TABLE IV

Molar Ratio of Components of Ethereal phases for Various Initial Concentrations of Ferric Chloride (Initial: 8.23 M HCl, and Volume Ratio $H_2O/IPE = 1.25 : 1$)

Initial concn. $FeCl_3$	Heavy ether phase $FeCl_3 : IPE : H_2O : HCl$	Light ether phase $FeCl_3 : IPE : H_2O : HCl$
0.05	1 : 10.1 : 6.8 : 1.3	1 : 14.7 : 6.2 : 0.8
0.10	1 : 11.9 : 7.0 : 1.4	1 : 11.7 : 5.9 : 1.1
0.17	1 : 12.0 : 6.3 : 1.2	1 : 11.9 : 5.5 : 1.2
0.40	1 : 10.6 : 5.4 : 1.2	— — — —
0.62	1 : 8.0 : 5.0 : 1.3	— — — —
0.99	1 : 4.7 : 3.9 : 0.9	— — — —
1.48	1 : 4.0 : 3.4 : 0.9	— — — —
2.22	1 : 3.2 : 3.0 : 0.7	— — — —
2.96	1 : 2.8 : 2.9 : 0.8	— — — —

TABLE V

Molar Ratio of Components of Ethereal phases for Various Initial Concentrations of Hydrochloric Acid (Initial: 0.197 M $FeCl_3$, and Volume Ratio $H_2O/IPE = 1.25 : 1$)

Initial concn. HCl	Heavy ether phase $FeCl_3 : IPE : H_2O : HCl$	Light ether phase $FeCl_3 : IPE : H_2O : HCl$
4.94	— — — —	1 : 327 : 7.5 : 0.8
6.18	— — — —	1 : 22 : 7.4 : 1.0
7.82	1 : 14.5 : 2.3 : 1.2	1 : 90 : 7.3 : 1.4
8.65	1 : 10.5 : 7.9 : 1.8	1 : 154 : 6.8 : 1.2
9.06	1 : 9.1 : 9.3 : 2.3	1 : 225 : 2.2 : 5.9
9.88	1 : 7.6 : 15.8 : 3.8	— — — —
10.29	1 : 5.1 : 21.2 : 2.6	— — — —

TABLE VI

Distribution of Iron (III) Extracted by Isopropyl Ether from Hydrochloric Acid (Initial: 0.197 M $FeCl_3$, and Volume Ratio $H_2O/IPE = 1.25 : 1$)

Initial concn. HCl (mol.)	Volumes of phases (ml.)		$\frac{Fe_{he}}{Fe_{aq}}$	$\frac{Fe_{he}}{Fe_{le}}$
	heavy	light		
10.29	5.4	—	30.8	—
9.88	8.2	—	20.8	—
9.06	9.7	14.2	1547.5	26.7
8.65	9.8	15.5	1966.6	13.1
8.23	11.0	16.9	1806.6	7.6
7.82	11.8	17.6	4600.0	6.0

he — heavy ether phase
le — light ether phase
aq — aqueous phase

The appearance of the third phase is practically independent of the ferric chloride content if the concentration of hydrochloric acid is above 7 *M* (Table II). From the results of analyses of both of the ether phases expressed as molar ratios of the components (Tables IV and V) some conclusions are obtained which are not in agreement with discussion reported in ref. 3, 6 and 23. According to several earlier publications of Myers *et al.*[6,23] the heavier ether phase (third phase) contains more water per iron than does the lighter phase, while the data reported by Laurene *et al.*[3] show that the third phase has a mole ratio of water to iron of less than five. Our results (Tables I and II) show that molar ratios of the components in the third phase depend on the initial concentrations of ferric chloride and hydrochloric acid. For various initial compositions of the systems the mole ratio of water to iron may be more than five in both the ether phases. The mole water per mole iron ratio in the third phase may also be greater or less than it is for the light ether phase.

The mole ratio of water per iron in the third phase increases with an increase in the initial concentration of hydrochloric acid or with a decrease in the initial concentration of ferric chloride. When the concentration of hydrochloric acid is constant the increase in the concentration of iron is accompanied by a decrease in the molar ratio of water per iron. According to our results (Figs. 5, 6, 7 and 8) it can be concluded that the region of the co-existence of three phases is connected to two-phase regions near the tetrahedron edge representing the system: H_2O-IPE. The third phase has not a constant composition and depends on the initial composition of the system (Table IV and V).

The fact that most of the extracted iron is accumulated in the third phase is very interesting from the point of view of separation (Table VI). The best separation of the iron could be obtained for 9.06 *M* hydrochloric acid since the lowest concentration of iron occurs in the aqueous and light ether phases.

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IZVOD

Ispitivanje sistema: ferum(III)klorid-hidrogen klorid-voda-izopropilni eter

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Izvršena su ispitivanja heterogenog ekstrakcijskoga sistema: ferum(III)klorid-hidrogen klorid-voda-izopropil eter kod 20^{0}C.

Prethodno su određene promjene volumena faza u ovisnosti o promjeni količine pojedine od komponenata sistema.

Analitičkom metodom su određeni ravnotežni sastavi faza četverokomponentnog sistema za različite početne koncentracije solne kiseline i ferum(III)klorida. Na osnovu tih rezultata su prikazane ortogonalne projekcije tačaka koje predstavljaju sastave faza u tetraedarskom prostoru.

Konačno se diskutiraju rezultati analiza i uvjeti prijelaza dvofaznog u trofazni sistem i obrnuto.

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